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# Optical and thermal properties of SiN<sub>x</sub> for MO disks

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## ABSTRACT

SiN<sub>x</sub> films were prepared by rf reactively sputtering. The refractive index of SiN<sub>x</sub> films was affected by total pressure and sputtering power. When the total pressure increased, the refractive index decreased. The reduction of sputtering power showed similar effect to raise the total gas pressure. The residual stress and roughness of SiN<sub>x</sub> films depended on the total pressure, sputtering power, and the thickness. The thermal cycles may result in irreversible change of residual stress of SiN<sub>x</sub> film. The magnetic properties of TbFeCo depended on the residual stress and roughness of SiN<sub>x</sub> in the trilayer SiN<sub>x</sub>/TbFeCo/SiN<sub>x</sub> samples. The coercivity of TbFeCo was enhanced in the samples with SiN<sub>x</sub> films having low stress and large roughness.

**Keywords:** SiN<sub>x</sub> films, TbFeCo films, refractive index, magnetic properties, residual stress, roughness, MO.

## 1. INTRODUCTION

Amorphous rare earth-transition metal (RE-TM) films are commercially used for the magneto-optical (MO) recording medium<sup>1,2</sup>. However, the rare earth elements can be easily oxidized. Protecting dielectric films are needed to inhibit the corrosion. Amorphous SiN<sub>x</sub> films were commonly used in MO disks<sup>1,2</sup> and their optical properties were studied. Other dielectric films, such as AlSiN, AlSiON and SiO<sub>2</sub>, were studied for altering the optical properties and for improving the protection capability<sup>2</sup>. Amorphous SiN<sub>x</sub> films may have large residual stress under certain deposition conditions<sup>3,4,5</sup> which may accompany with the change of the optical properties. RE-TM films were known to have large magnetostriction coefficients<sup>6,7,8</sup>, so the stress state may affect their magnetic properties. As the result, the stress state in dielectric films may affect the magnetic properties of MO films. In this paper, SiN<sub>x</sub> dielectric films were deposited by rf reactively sputtering, and the effect of deposition conditions on the index of refraction was studied. In addition, the dependence of stress state and roughness of SiN<sub>x</sub> films on the deposition conditions was investigated. The correlation of magnetic properties of MO (TbFeCo) films with the SiN<sub>x</sub> properties is reported. Since the writing process of MO recording is of a thermal writing process, the thermal cycles are imposed to MO disks. Due to the difference of thermal expansion coefficient between films and substrates, the thermal stress may be developed during the thermal cycles. The thermal stress and its effect on the magnetic properties of MO films are discussed.

## 2. EXPERIMENTAL PROCEDURE

SiN<sub>x</sub> films, deposited in a planetary sputtering system, were reactively sputtered on Si (100) substrates by using a Si target and a rf power supply. The total working pressure, the sputtering power and the thickness were varied during the deposition to manipulate the optical properties of SiN<sub>x</sub>. The trilayer of SiN<sub>x</sub>/TbCoFe/SiN<sub>x</sub> was sequentially deposited on Si (100) substrates without breaking vacuum. An alloy target with composition TbFeCo(Tb:20 atomic%, Fe:72 atomic%, Co: 8 atomic%) was used for TbFeCo film. The base pressure before depositions was 3\*10<sup>-7</sup> torr. The deposition conditions for the TbFeCo film were the following: dc power=200W and total pressure=5mTorr. After depositions, x-ray diffraction was used for structural characterization, and atomic force microscope (AFM) was used for surface roughness measurement. The composition of films was determined by using Rutherford backscattering (RBS). The index of refraction *n* of SiN<sub>x</sub> and the film thickness were measured by *n* & *k* analyzer with the light wavelength of 680 nm. The bending beam method<sup>9</sup> was carried out to measure thermal stress of SiN<sub>x</sub>. The temperature was increased from 25 °C to 400 °C, and the thermal stress

was in-situ determined by the curvature of the samples. The residual stress in  $\text{SiN}_x$  after depositions was measured by using wafer curvature. The hysteresis loops of the trilayers were measured by perpendicular magneto-optical Kerr effect (PMOKE) at room temperature.

### 3. RESULTS AND DISCUSSIONS

In order to reduce noise,  $\text{SiN}_x$  and TbFeCo need to be amorphous. From x-ray  $\theta$ - $2\theta$  scans and grazing angle scans,  $\text{SiN}_x$  and TbFeCo were verified as amorphous phases, as shown in Fig. 1. After thermal cycles up to 400 °C, both films still maintained their amorphous phases.

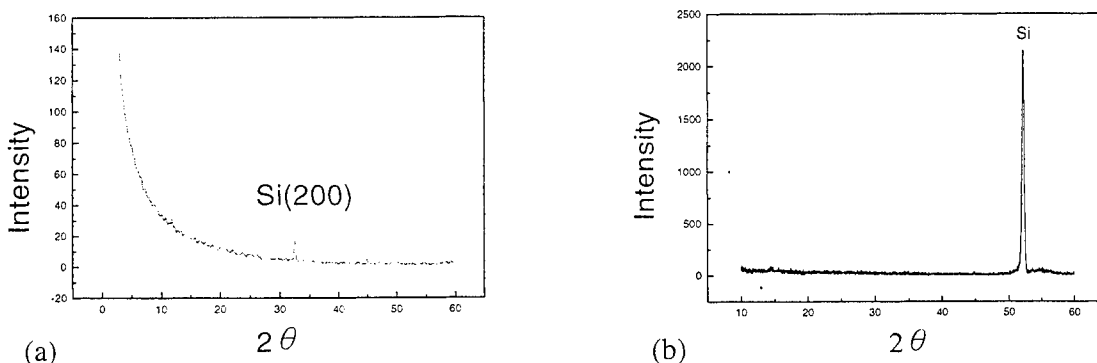


Fig. 1 X-ray scans of 90nm  $\text{SiN}_x$ /30nm TbFeCo/90nm  $\text{SiN}_x$  trilayers. (a)  $\theta$ - $2\theta$  scan (b) grazing angle scan.

The dependence of refractive index  $n$  on the total gas pressure is shown in Fig. 2. The  $n$  value decreased from 2.6 to 1.6 with increasing total pressure from 3 m torr to 15 m torr. When the total gas pressure increased during depositions, the kinetic energy of sputtered atoms arriving substrate surface decreased due to the scattering with ionized plasma. Consequently, the stacking density of  $\text{SiN}_x$  films decreased with increasing total pressure. Refractive index is equal to the ratio of the light speed in vacuum to the speed in the medium ( $n=c/v$ ). The light speed is faster in the less dense medium, and therefore the  $n$  value is smaller. In order to confirm the film density,  $\text{SiN}_x$  was put into  $\text{H}_3\text{PO}_4$  (85%, 180°C) solution, and the rate of wet etching is shown in Fig. 3. When the total pressure increased, the etching rate increased, consistent with the decrease in the film density.

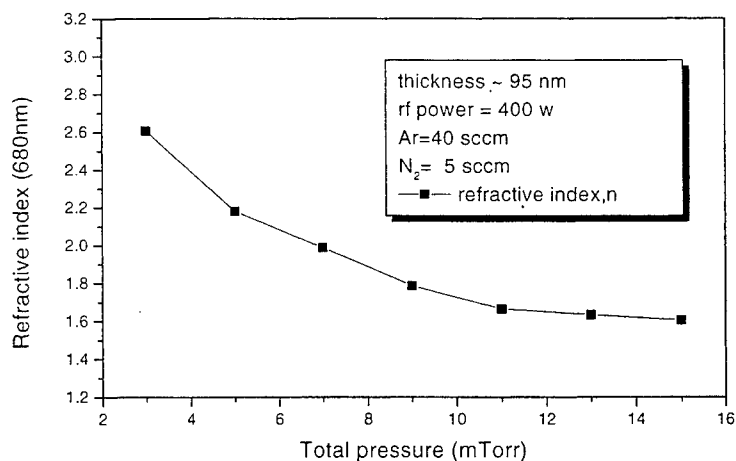


Fig. 2 Dependence of refractive index  $n$  of  $\text{SiN}_x$  films on the total gas pressure. The deposition parameters of  $\text{SiN}_x$  films are shown in the inset.

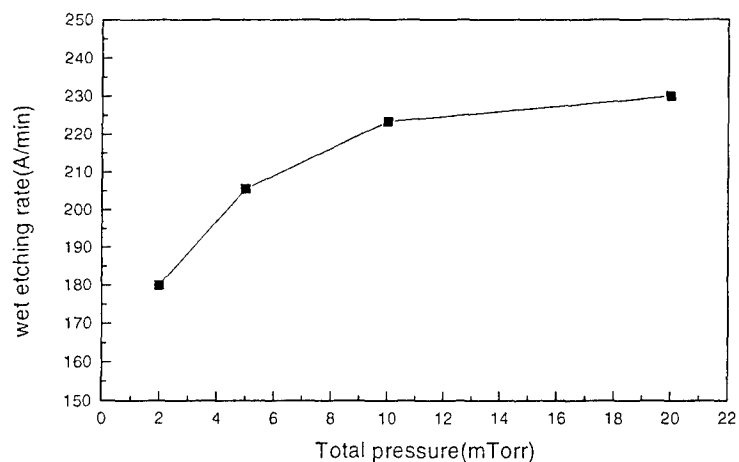


Fig. 3 Dependence of wet etching rate of  $\text{SiN}_x$  films on the total gas pressure. The etching solution is  $\text{H}_3\text{PO}_4$  (85%, 175°C) .

The dependence of the residual stress on the total pressure is shown in Fig. 4. At low pressure (2 m torr), the atomic pinning effect may be dominated, so the  $\text{SiN}_x$  film exhibited compressive stress<sup>5</sup>. When the total pressure increase, the atomic pinning effect is suppressed, and hence the compressive stress was reduced. When the total pressure was further increased, the film might become porous and the model of grain boundary relaxation can be applied<sup>5,10,11</sup>. The films deposited at high total pressure showed tensile stress. The surface roughness (root-mean-square, r.m.s. roughness) was significantly increased with increasing total pressure, shown in Fig. 5. This relationship was commonly observed in other dielectric films. When the total pressure increased, the kinetic energy of sputtered atoms arriving substrates reduced so they could not land on the proper sites to reduce the roughness.

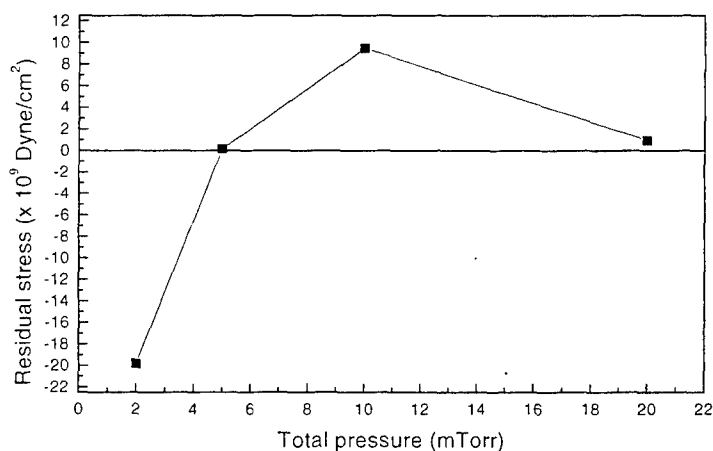


Fig. 4 Variation of residual stress of  $\text{SiN}_x$  films with the total gas pressure.

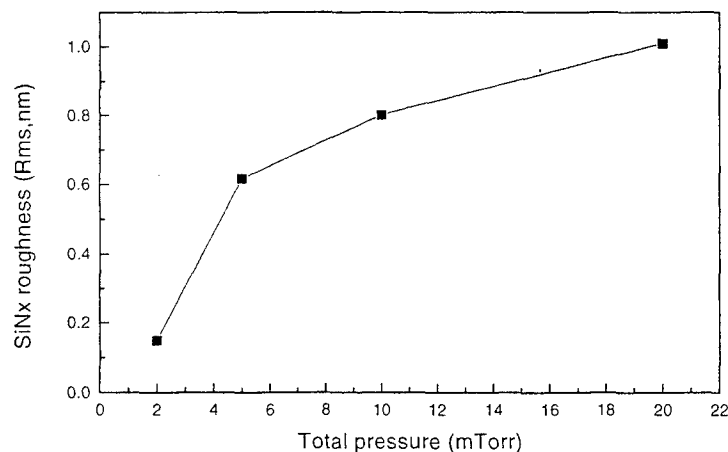


Fig. 5 Variation of root-mean-square (r.m.s.) roughness of  $\text{SiN}_x$  films on the total gas pressure.

The variation of  $n$  with the sputtering power is shown in Fig. 6. The  $n$  value was reduced in the films deposited at lower rf power. When sputtering power reduced, the kinetic energy of sputtered atoms arriving the substrate surface was reduced. The effect is similar to the increase in the total pressure, resulting in less dense film, and therefore a lower refractive index. The roughness decreased with increasing sputtering power, shown in Fig. 7., which resulted from the increase of kinetic energy of sputtered atoms: therefore, the film was smoother and denser for high sputtering power.

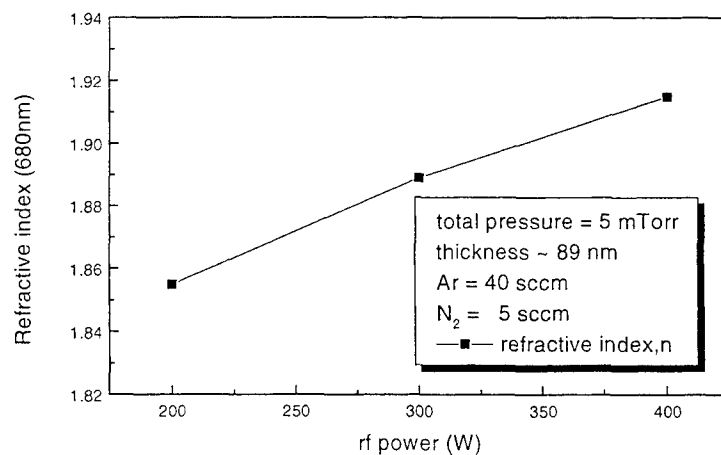


Fig. 6 Variation of refractive index  $n$  of  $\text{SiN}_x$  films with the sputtering power. The deposition conditions are shown in inset.

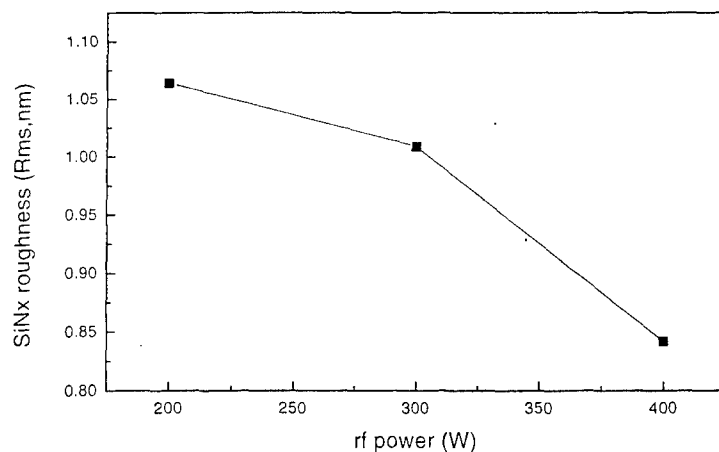


Fig. 7. Dependence of r.m.s. roughness of  $\text{SiN}_x$  films on the sputtering power.

The refractive index of  $\text{SiN}_x$  is insensitive to the film thick ranging from 50 nm to 400 nm, as shown in Fig. 8. In contrast, the residual stress and the surface roughness were strong functions of the film thickness. The residual stress and roughness increased significantly with increasing thickness, as shown in Fig. 8. and Fig. 9., consistent with other sputtered dielectric films. The  $\text{SiN}_x$  developed the columnar structure during the sputtering process, so the stress and roughness can be added up within each column. Consequently, both the stress and roughness increased when the films became thicker.

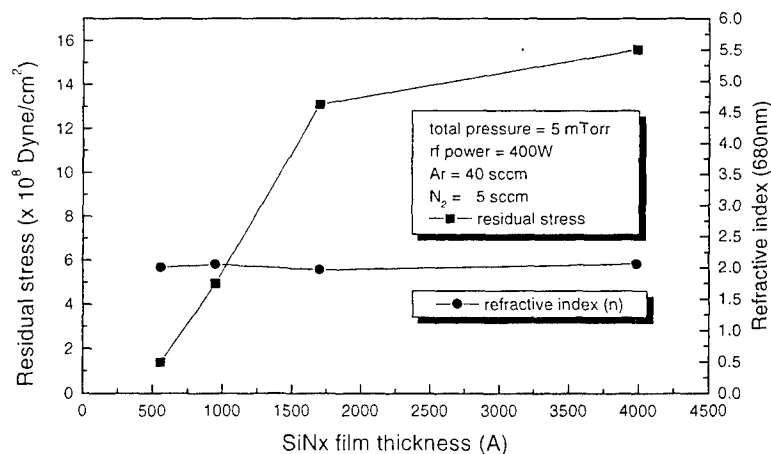


Fig. 8. Dependence of the refractive index and the residual stress and on the  $\text{SiN}_x$  film thickness. The deposition conditions are shown in inset.

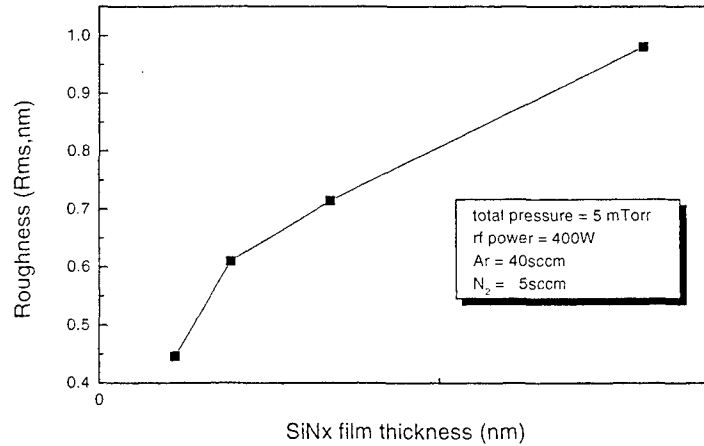


Fig. 9. Dependence of the r.m.s. roughness on the SiN<sub>x</sub> film thickness.

The measurement of thermal stress of SiN<sub>x</sub> films was carried out at the temperature ranging from 25 °C to 400 °C. During the cooling process, the residual stress became toward tensile stress ,as shown in Fig. 10. During the heating process, the stress almost unchanged. After thermal cycles, residual stress exhibited irreversible change. For the specific sample shown in Fig. 10., after two thermal cycles, the stress changed the sign, from compressive stress to tensile stress. The irreversible change of the stress may result from two reasons: difference of thermal expansion coefficient between the film and substrate and (or) change of local atomic arrangement. The detailed study of the stress change due to the thermal cycles is currently under investigation.

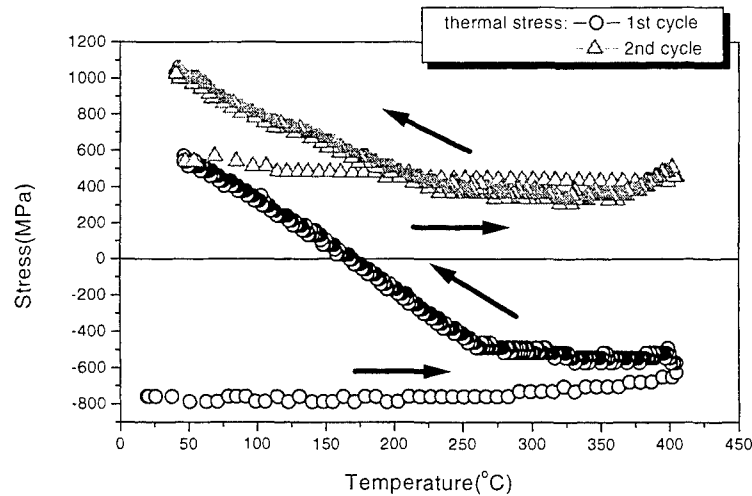


Fig. 10. Relationship between the residual stress of SiN<sub>x</sub> films and temperature. Residual stress decreased during the cooling process and almost unchanged in the heating process. After two thermal cycles, the residual stress changed from compressive to tensile.

The hysteresis loop of the trilayer sample 90 nm SiN<sub>x</sub>/40 nm TbFeCo/90 nm SiN<sub>x</sub> is shown in Fig. 11. The perpendicular anisotropy was observed with the coercivity of 11000 Oe. Several deposition parameters were varied to manipulate the stress state and roughness of SiN<sub>x</sub> films, but meanwhile the deposition conditions for TbFeCo films were maintained to investigate the effect of SiN<sub>x</sub> properties on the magnetic properties of TbFeCo films. The summary of the r.m.s. roughness, residual stress and coercivity of different samples, is shown in Table. 1. The sample A has almost zero residual stress and the highest roughness, showing the highest perpendicular coercivity (~11000 Oe). The hysteresis loop of sample A is shown in Fig. 11. Comparing sample B and C, the magnitude of r.m.s. roughness of SiN<sub>x</sub> films is about the same, but the tensile stress in sample B is almost 100 times larger than in sample C. The coercivity of sample B is much less than that of sample C. Sample D and E have similar compressive stress in SiN<sub>x</sub> films, but have different r.m.s. roughness. Large roughness in sample D seemed to enhance the coercivity. Based on these results, the roughness and stress in SiN<sub>x</sub> may play important roles to determine the magnetic properties of TbFeCo films. It is plausible that the residual stress in SiN<sub>x</sub> may affect the stress state in TbFeCo films. Because of high magnetostriction coefficient in TbFeCo films, the stress state can significantly affect the anisotropy of TbFeCo, and hence the coercivity. Roughness in SiN<sub>x</sub> films may provide extra pinning sites in TbFeCo films. Consequently, the coercivity is enhanced in the samples with large roughness.

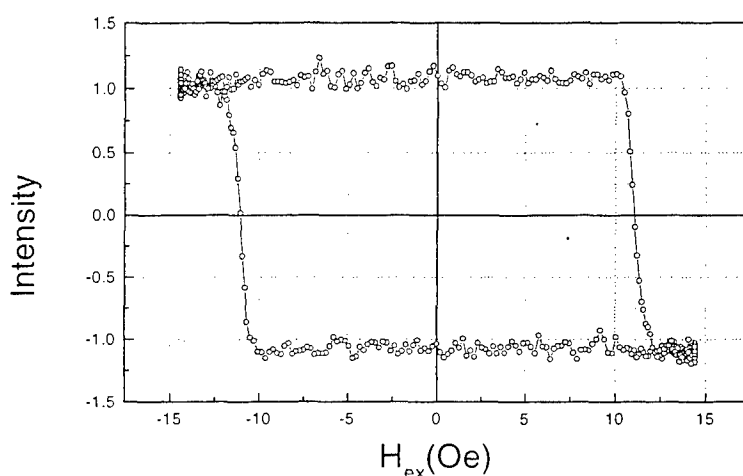


Fig. 11 Hysteresis loop of SiN<sub>x</sub>/TbFeCo/SiN<sub>x</sub> measured by PMOKE at room temperature. The residual stress of SiN<sub>x</sub> in the sample is almost zero and the r.m.s. roughness of SiN<sub>x</sub> is 10 Å.

Table 1 Summary of r.m.s. roughness, residual stress, and coercivity of trilayer SiN<sub>x</sub>/TbFeCo/SiN<sub>x</sub> samples. The r.m.s. roughness and residual stress are the values in SiN<sub>x</sub> films.

	Rms	Residual stress dyne/cm <sup>2</sup>	Hc(kOe)
Sample A	10.093Å	~ 0	11.0705
Sample B	8.03Å	9.48E+09	6.63148
Sample C	8.42Å	1.52E+08	10.8095
Sample D	7.508Å	-7.58E+09	10.05418
Sample E	1.5Å	-1.98E+10	3.4227



#### 4. CONCLUSIONS

SiN<sub>x</sub> films were prepared by rf reactively sputtering. The refractive index of SiN<sub>x</sub> films were affected by total pressure and sputtering power. When the total pressure increased, the kinetic energy of sputtered atoms arriving substrates decreased. Consequently, the films became less dense, so the refractive index decreased. The reduction of sputtering power showed similar effect to raise the total gas pressure. The residual stress and roughness of SiN<sub>x</sub> films depended on the total pressure, sputtering power, and the thickness. By adjusting the deposition parameters, the residual stress and roughness of SiN<sub>x</sub> films can be manipulated. The thermal cycles may result in irreversible change of residual stress. The magnetic properties of TbFeCo depended on the residual stress and roughness of SiN<sub>x</sub> in the trilayer SiN<sub>x</sub>/TbFeCo/SiN<sub>x</sub> samples. The coercivity of TbFeCo was enhanced in the samples with SiN<sub>x</sub> films having low stress and large roughness.

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